



DECLARATION UNDER 37 C.F.R. 1.132

I, Dr. Michael A. Bryner, do hereby declare as follows:

1. I was granted a Bachelor of Science degree in Chemical Engineering from Clarkson University in 1966; a Master of Science in Chemical Engineering from Clarkson University in 1970; and a Doctor of Philosophy in Chemical Engineering from Clarkson University in 1973. My dissertation was entitled "A Unified Statistical Analysis of Two-Phase Flows Through Porous Media". I am the holder of six patents and most recently a coauthor of a peer reviewed paper entitled "Designing Nonwovens to Meet Pore Size Specifications" (Journal of Engineered Fibers and Fabrics, Volume 2, Issue 1 – 2007, pp. 1-15)

2. I am the inventor of the claimed invention this U.S. Patent Application No. 10/664,708, and worked for the assignee, E.I. du Pont de Nemours and Company between 1973 and 2005, and am an expert in fiber spinning from the melt, via flash spinning from polymer solution, and by electrospinning. I have expertise as well in the nature and properties of porous media and in the relationship between the structure and properties of nonwoven fabrics. Presently, following retirement in 2005, I am a limited service employee for DuPont Nonwovens Business Unit.

3. I have read, understood and considered the patent examiner's rejections in the outstanding Office Action, issued 5 June 2007, under 35 U.S.C. § 103(a), as well as the prosecution history of the application in general.

4. I disagree with the Examiner's conclusion that the claims were anticipated or would have been obvious to the skilled artisan in view of the cited references at the time of my invention thereof. I express my concerns below.

5. General State of the Prior Art

The prior art can generally be characterized as follows:

- A) The prior art cited in the rejections is almost exclusively devoted to exploring the use of nanofibers in filtration applications.
- B) The prior art overwhelmingly shows and leads to the expectation that application of nanofibers to a substrate significantly lowers composite fabric air permeability. I quoted this finding directly to the Examiner from one of the Natick – Gibson papers during the personal interview conducted with the Examiner on March 6, 2007. The underlying reason for this is demonstrated in the present specification.
- C) Nowhere in the prior art is there a conception of achieving both high liquid barrier (hydrohead) AND high air permeability (Frazier) via incorporating nanofibers into composites.

- D) Nothing in the nanofiber prior art recognizes the problems of interplay between fiber strength, basis weight and substrate properties which can limit hydrohead for weak fibers. (No one ever made fabrics before with hydrohead high enough to demonstrate the problem).
6. The invention of this application rightly falls within the general art of product development using composite nonwoven fabrics. In my opinion, the level of ordinary skill in the art is represented by one having attained a bachelor of science degree in chemical, mechanical, or textile engineering or the equivalent and at least three years of work experience in this field. However, the ordinary skills in this art do not generally include proficiency in the electrospinning of fine fibers which was the only practical way of producing polymeric nanofibers for the invention.

The electrospinning of polymeric nanofibers is itself a separate art. In my opinion, the level of ordinary skill in this art is represented by one having attained a bachelor of science degree in engineering or science and at least one year of work experience in this field. The ordinary skills in this art do not generally include proficiency in developing composite nonwoven fabrics.

7. Distinctions between nonwoven filtration and barrier fabrics

- 7.1 The Examiner seems to consistently hold that the properties of nonwoven fabrics created for filtration end-uses are the same as or predictive of fabrics created to act as liquid barriers. That this is not the case was discussed with the Examiner in the personal interview, conducted on March 6, 2007.
- 7.2 During the personal interview we discussed that the requirements for high liquid barrier performance were more restrictive than for air filtration performance. I pointed out that filtration performance can be excellent even though there may be fiber maldistributions and defects which create holes – e.g., pinholes, in the fabric. However, a fabric with such pinholes would have low hydrohead. I explained that a hydrohead measurement is of its nature also a defect measurement. Achieving high liquid barrier in terms of high hydrohead imposes a different, more stringent set of requirements on fiber distribution and defects.
- 7.3 There is also the issue of polymer/fluid interaction. Wettability is of no concern or issue with regard to the performance of air filtration fabrics, but it is of critical importance for liquid barrier products. High liquid barrier is not possible if fibers are wetted by the liquid.
- 7.4 The development of the model in the present specification shows that to a first order, hydrohead is not dependent on fabric basis weight. In contrast,

it is well known in the art that filtration performance is a direct function of basis weight. Filtration performance is increased by increasing basis weight and decreased by decreasing basis weight.

- 7.5 The bottom line is that there are many more factors and requirements for a fabric to achieve high liquid barrier as opposed to high air filtration efficiency. The reality is that one cannot reliably infer liquid barrier performance from a reference base of filtration performance. "Improved barrier" as cited in the results and objectives of much of the prior art where barrier is conceived and measured in terms of filtration performance cannot be presumed to imply improved liquid barrier. One skilled in the art would know that this extrapolation is not reliable.

8. Review of the Doshi Reference

- 8.1 Doshi's results show improved filtration performance of a nanofiber/substrate composite over that of the substrate alone. But the results of Table 2 reveal that improved hydrohead is not predicted by the application of nanofibers alone. (Note that hydrohead for the spunbond substrate alone is not listed, presumably because it is too low to be measurable). Application of nanofibers to this substrate resulted in an increase in hydrohead to 15.0 mbar. However, application of nanofibers to the melt blown substrate actually lowered the hydrohead by more than a factor of 3. What's more, the hydroheads measured on the nanofiber composites were essentially the same, regardless of the properties of the substrate and in spite of the fact that the nanofiber web basis weight on one sample was double that of the other.
- 8.2 Lastly Doshi's results clearly show, as does all of the prior art, that application of nanofibers to a substrate decreases air permeability very significantly.
- 8.3 Summary: Doshi's paper teaches the utility of applying nanofibers to substrates to improve filtration performance, but in no way can one infer anything about liquid barrier performance. This demonstrates the point made in paragraph 7, above.

9. Examiners Assertions re: Doshi

In the outstanding Office Action, the Examiner states:

Although Doshi does not appear to explicitly teach the claimed hydrohead value or Frazier permeability, it is reasonable to presume that said limitations are inherent to the invention. Support for said presumption is found in the use of similar materials (i.e. nylon nanofiber barrier layer) and in the similar production steps (i.e. bonding to a substrate layer) used to produce the nonwoven

fabric. The burden is upon the applicant to prove otherwise. In addition, the claimed limitations are readily obtainable through routine experimentation with variables such as fiber material, fiber diameter, basis weight, solids fraction, maximum pore size, and because it is understood by one of ordinary skill in the art that discovering an optimum value of a result effective variable involves only routine skill in the art. It is noted that Doshi specifically teaches that the material is made in order to create a barrier layer with improved hydrohead. (Emphasis added).

- 9.1 Initially, the Examiner's statement in the last sentence above is not correct. Nowhere in Doshi's paper does he even mention the objective or the utility of applying nanofibers to a substrate to improve hydrohead. He expressly addresses the problem of the filtration of fine particles and his conclusion demonstrates the utility of applying nanofibers to improve filtration performance. Doshi specifically states in the third introductory paragraph:

In this article we focus on providing a nanofiber-based solution to a significant problem facing the manufacturing process industries today. The problem is that of filtration of fine particles from industrial effluents.

- 9.2 The Examiner's assertion as to the inherency of Doshi's Frazier and Hydrohead values is without merit. Under "Results and Discussion", Doshi states:

Fabric resistance to water penetration was not increased as was expected with the incorporation of nanofiber web. It was observed (Figure 8) that the nanowebs had very high absorbency (ASTM D 461), and because of this, the trend in observed hydrohead is not consistent with the decrease in air permeability. (Emphasis added).

By the logic in paragraph 7 above, Doshi's results for filtration cannot be construed to "inherently" address and include the issues, conditions, and/or results pertaining to the high hydrohead of the invention. This is particularly evident by my results set forth in the present specification, wherein I achieved 10X the hydrohead levels disclosed by Doshi, and further, in combination with high air permeability.

Doshi does not anticipate the invention of the present application in terms of either the concept and/or the need for this combination, or the significant obstacle of the barrier fiber/substrate/fiber strength relationship that must exist for a 10X increase in hydrohead. In fact, the actual measurements on his fabrics specifically demonstrate that the composites he made would not satisfy the objectives of my claims.

9.3 *"Routine Optimization"--Level of Ordinary Skill in the Art*

(a) I take particular issue with the Examiner's suggestion that my invention would have been obtainable by the mere "routine optimization" of various known-in-the-art variables for nonwoven fabric design and production.

(b) The techniques and methodologies for experimental optimizations by those of ordinary skill in the art, e.g. as taught by Six Sigma, are typically limited to linear interrelationships between variables. For nonlinear systems the ordinary skilled artisan would approximate real systems by using a prescribed base point and a local linearization of variable dependencies around that base point. This means that the methodologies and the results from using them are valid near the conditions chosen for linearization, that is, within the region of small changes from the base condition. Typically, then, optimizations by those of ordinary skill in the art of nonlinear systems by the techniques common in the art can reasonably be expected to be successful for adjustments of variables and resulting properties not too far-removed from the base.

(c) In contrast, as I have developed in the specification of this Patent Application No. 10/664,708, the interrelationships among the fabric variables governing the balance of hydrohead and Frazier are demonstrably nonlinear. In Claim 14, these relationships have been approximated with a mathematical fit of the model of the specification. The nonlinearity of the fit and of the property interrelationships is self evident.

(d) The Examiner holds that since all nanofiber composite fabric properties derive from the same set of variables, and since Doshi has made nanofiber composite fabrics, the properties claimed in the invention are held to be inherent in Doshi's fabrics. Further, if the properties of the Doshi fabrics are not the same as claimed in the invention, the Examiner argues, that since Doshi uses nanofibers of the same size I claim and barrier layer basis weights in the range I claim on substrates as I claim, that the properties I claim are at least close enough in the Doshi composites to invite one skilled in the art to attain them through simple adjustments- "optimization" - of key variables.

(e) By the description of the methodology above, this would be a reasonable argument if the invention were to claim a slight increase in hydrohead over Doshi, and a slight increase in Frazier, for example, say a hydrohead of 30-40 bar with a Frazier of 1 to 5. But as defined in Claim 1, the invention comprises an order of magnitude – 10X shift in the hydrohead base point described by Doshi, without any loss of Frazier over Doshi. Such an order of magnitude change so far removed from the

Doshi base point is not a reasonable a priori expectation for the application of normal linear optimization techniques.

9.4 *"Routine Optimization"--That which would be necessary to modify the prior art in the manner of the present invention*

(a) The straight forward optimization envisioned by the Examiner as "easily achievable by one skilled in the art" implies having or developing ways of measuring and controlling each of at least seven variables. It further implies having or developing methods of fabricating composite webs incorporating the specified variables, and the methods of measuring the properties of interest, namely air permeability and hydrohead.

(b) At a minimum, the air permeability and hydrohead properties of a nanofiber-based composite sheet depend on the following barrier layer variables, as developed in the present specification:

(1) Solidity--can practically vary from 10% to 90% and was known at the time of my invention to be controlled by the level of calendering (i.e. temperatures and pressure) of a web--however, it was questionable whether nanofiber webs could be successfully calendered in the extreme without degrading into films, due to the extremely small fiber diameters. Moreover, at the time of the invention, the square yardage of nanofiber-based fabrics required for typical calendering tests envisioned by the Examiner was not generally available.

(2) Fiber size (diameter)--can practically vary from 50 nm to 1000 nm, but actual control of fiber diameters by electrospinning has been the subject of continuing research and speculation. For each polymer/solvent system electrospun, the generation and control of fibers of a specific submicron size is itself an "optimization" process with its own set of variables.

(3) Fiber size distribution--average of fiber diameters and their distribution about the mean, varying between the practical limits set forth above in item (2), but difficult to measure or control at the time of filing of the present application.

(4) Spatial distribution of fibers--degree of clumping and randomness of fiber axis direction on the fine scale --no suitable direct measurement (can be inferred from hydrohead). Web uniformity on a slightly larger scale -- difficult to measure locally.

(5) Fiber hydrophobicity--can be affected by polymer type and/or polymer surface treatment--however, few "hydrophobic" polymers are electrospinnable into nanofibers due to lack of suitable

polymer/solvent systems and the generally non-polar nature of most hydrophobic polymers. Optimization using hydrophilic polymers such as the nylon in Doshi's fabric would require hydrophobic chemical treatments or coatings as post treatments. Such treatments or coatings introduce a number of additional variables and issues, for example, the nanofibers must be treated uniformly along their length, the treatment/coating must not block the pores which are already small, and the entire process of post treatment must not break fibers or disturb the spatial distribution of fibers in the structure.

(6) nanofiber web basis weight--almost infinitely variable, but for practical purposes, between about 0.5 and 20 gsm by electrospinning over very small areas.

(7) nanofiber layer strength in relation to the support layer pore size--no suitable measurement technique for this relationship at the time of filing of the present application, but a variety of different support layers could be tested. Note, per the discussion of the specification of this Patent Application No. 10/664,708, a priori selection of support layers with pore sizes "too large" to adequately support the weak nanofiber layer would result in fiber breakage under load leading to erroneous conclusions about the relationship between hydrohead and fabric variables and about the potential for achieving the high hydrohead objective.

(c) Assuming that one of ordinary skill in the art could over-come the stated technical drawbacks in items 9.4(b)(1)-(b)(7) above, he would be presented with an enormous task to determine the interrelationships between the above variables and "optimize" both hydrohead and permeability experimentally. For example, choosing just three levels of each variable would require $3 \times 3 \times 3 \times 3 \times 3 \times 3 \times 3 = 2187$ experiments to fully explore all combinations of variables. Statistical techniques exist to identify the most significant variables and to reduce the number of experiments, but the methodologies are complicated by the unusual requirement to simultaneously "optimize" two fabric properties which are in opposition to one another. I submit that such techniques and methodologies are generally outside of the level of ordinary skill in the art.

(d) The effort to electrospin only the two polymers reported in the application, to develop suitable means to uniformly deposit nanofibers onto substrates, to develop calendering methods, and to measure fiber and fabric properties all comprising the "optimization" reported in the application required just over 100 experiments and an estimated 1000 man hours. This was in spite of having the mathematical model to define the effects of key variables and to guide the effort.

(f) I submit that amount of experimentation which would be necessary "optimize" the stated variables in the manner suggested by the Examiner, represents an undue level of experimentation and, per paragraph 6 above, a skill level outside and above that ordinarily found in the art of composite nonwoven fabrics and/or that of electrospinning of polymeric nanofibers. In fact, for this reason I took a different approach, that of developing the model presented in the Specification of the application.

10. Unexpected Results over Closest Prior Art of Doshi

10.1 Even if, for the sake of argument, one skilled in the art could reasonably have been expected to be able to optimize both hydrohead and air permeability together, nothing in the prior art would lead to the expectation of the unique combinations of hydrohead and Frazier demonstrated in the claimed invention.

10.2 In Example 10 of this application, I report a fabric with a Frazier permeability of $11.2 \text{ m}^3/\text{m}^2\text{-min}$ ($36.7 \text{ ft}^3/\text{ft}^2\text{-min}$) with a hydrohead of 131 cmwc (128 mbar). This is 22X the air permeability and almost 9X (8.9X) the hydrohead of Doshi's best sample. My highest barrier sample at 399 cmwc (391 mbar) has ~30X the hydrohead of Doshi's samples and it still has higher Frazier permeability – $0.7 \text{ m}^3/\text{m}^2\text{-min}$ ($2.3 \text{ ft}^3/\text{ft}^2\text{-min}$), i.e., still 1.4X Doshi's sample. A priori expectation of these results based on the prior art is unreasonable.

11. Long Felt Need/Failure of Others

11.1 I submit that at the time of the invention there were no known fabrics of the type of the invention having hydrohead and air permeability in the range demonstrated and claimed herein. Further, I believe that the demonstration of this invention was the first such demonstration in recorded history of these particular combinations of high hydrohead and high air permeability using polymeric nanofiber based fabrics.

11.2 Additionally, in the time since this application was filed, in spite of the commercial desirability of this combination of properties as set forth in this application, and in spite of the growth in the field of nanofiber based fabric offerings, I know of no other disclosure outside of DuPont wherein a polymeric nanofiber composite has achieved the unique balance of properties achieved and claimed in this application.

11.3 I submit that this is evidence of long felt need for my invention, in combination of the failure of others to achieve the goals set forth in the claims.

12. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to

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be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Michael A. Bryner
Michael A. Bryner

8/30/07
Date